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DATE: July 6, 1995
SUBJECT: Information Update #13

1. Q. Is "Pyrex Accelerated One-Step Extractor Concentrator" manufactured by Corning approved for EPA Method 3520, the liquid-liquid extraction technique?

A. Yes. For pesticides and PCB's the extraction time is reduced from 18 hours to 5.5 hours and for semi-volatiles, the extraction time is reduced from 36 hours to 12 hours. However, the initial demonstration must be done for accuracy and precision to verify that this technique is equivalent to or better than EPA 3520. EPA recommends running seven replicate matrix spikes by both the techniques and calculating % RSD and the spike recoveries for verification.

2. Q. Is "ASE 200 Accelerated Solvent Extractor" manufactured by Dionex approved for the extraction of solid samples in place of EPA Method 3545?

A. No. The draft version of EPA 3545 is still in the process of printing and has not yet been released. This Method will not be approved for at least a year. The proposed EPA Method 3545 is an alternate technique to the soxhlet extraction, which uses pressure and temperature to speed up the extraction process. The extraction time is cut to 12-18 minutes. The solvent extractor manufactured by Dionex has to go through an EPA approval process, after the Method has been approved. However, this extractor can be used for non-compliance testing.

3. Q. Has Key Scientific Products Konfirm been approved for water coliform testing?

A. No. Konfirm should not be used for EPA mandated compliance monitoring.

4. Q. Can EPA Method 200.2 be used as a standard digestion procedure for ICP and GFAA metals analysis for drinking water?

A. Yes.

5. Q. When does the holding time for Volatile Organic Compounds end on the fourteenth day?

A. According to Jean Munch of Drinking Water EPA, the holding time for the Volatile Organic

Compounds analyses in water will end at midnight on the fourteenth day from the day of sampling. The desorption of the analytes must be completed and transferred to the chromatography column before midnight.

6. Q. Is it mandatory to use Fluorobenzene as the internal standard for EPA Method 524.2 or can it be substituted?

A. Fluorobenzene is chosen by EPA as the internal standard for Method 524.2 for two reasons;

- a) Fluorobenzene has 100% purging efficiency and hence is a more reliable compound to be used as an internal standard.
- b) Fluorobenzene is never found in the environment and, therefore, has no possibility of it being found as a contaminant.

Deuterated compounds can be used as an alternate internal standard but they are more expensive. If substituted, according to Jean Munch of EPA, there must be a good reason for it.

7. Q. Is it acceptable to drop some runs from the MDL study and how does the laboratory determine the spiking amount?

A. MDL study is a measure of precision over multiple days. To begin, determine the estimated MDL which is three or five times the instrumental noise. Spike at one to five times the estimated MDL (spiked level must be 0.5 ppb or lower for 502.2 and 524.2, according to Jean Munch of EPA, Drinking Water) to calculate the MDL. According to Jean Munch, 1.0 or 2.0 ppb is too high for spiking for the MDL study. The laboratory must be confident of detecting the standard that was used to generate the MDL at all times. The standard must be included in the calibration curve. The study must be done over several days. All the runs must be included in the study. It is all right to run more than seven replicates but all the runs must be included in the calculation unless there is an obvious reason for dropping the run/runs. For example, if the last two runs, out of a total of nine runs, were dropped due to unacceptable surrogate recoveries, the first seven runs can still be used to calculate the MDLs but the cause for the drift must be investigated and documented before running more samples.

8. Q. For EPA Method 1311, Toxicity Characteristic Leaching Procedure, is it required to complete the rotation as well as the filtration within 18 +/- 2 hours?

A. No. According to Ollie Fordham of Solid Waste USEPA, only the rotation has to be completed within 18 +/- 2 hours. The filtration has to be completed soon afterwards on the same working day.

9. Q. For metal analyses, can the spiking be done after the acidification of the TCLP extracts?

A. No. According to Ollie Fordham of Solid Waste USEPA, the TCLP extracts cannot be acidified before spiking unless there is a problem with the TCLP extracts (like being basic), that necessitates the acidification before spiking for getting better spike recoveries. In that case the laboratories should do the side-by-side study to demonstrate that the modification works.

10. Q. Is it required to use 5% hydrogen in argon gas mix during dry and char steps in 200.9 (Table 2, Revision 1.2, April 1991, Method 200.9)?

A. Yes, according to Ted Martin of Drinking Water USEPA. It corrects the chloride interference in the samples. The laboratories can continue to use other platform methods until they are withdrawn, if they do not wish to use hydrogen for 200.9.

11. As part of their permit requirement, the City of Phoenix specifies the following method to be used for sulfide analysis in waste water: Analysis must be done in accordance with Standard Methods, 18th edition, 4500-S², as it is the only method that specifically addresses the existence of interferences and how they must be dealt with (4500-S² C for sample pretreatment, 4500-S² D for colorimetric or 4500-S² E for titrimetric). EPA Methods 376.1 or 376.2 can result in both false highs and false lows.
12. The following should clarify the confusion among the laboratories regarding the requirement of digestion for arsenic and selenium analyses in drinking water, if the turbidity is less than 1 NTU. The digestion is not required for arsenic, selenium or any other metal analyses in drinking water if the turbidity of the acidified sample is less than 1 NTU except for mercury and the gaseous hydride analyses. The turbidity must be measured using an approved method and only after preservation is complete. Preservation is complete after the acidified sample has been held for 16 hours. Before sample processing is started, sample pH must be verified to be less than 2. If selenium is being analyzed using SM 3113B, when nickel nitrate is used as the matrix modifier, an appropriate volume of 30% hydrogen peroxide (2-mL of 30% H₂O₂ per 100 mL of sample or standard) must be added to both the calibration standards and sample prior to analysis.
13. The new requirement for acceptance limits for Instrument Performance Check (IPC) in 200.7 (Revision 4.4, May 1994) is +/- 10%. This has been changed from +/- 5% from the previously approved version of 200.7, Revision 3.3, April 1991.
14. The deadline for WP034 was extended until June 30, 1995. This Office received the information late. WS036 is being shipped and the deadline for mailing the results is August 21, 1995. If the laboratories do not receive the shipment by July 5, 1995, call Natalie Murff at 513-569-7196. If additional proficiency sample vials are needed for a particular test, for any of the studies, fax the necessary information (your laboratory identification number, the sample being requested) to Natalie Murff, at 513-569-7115.

Your laboratory is required to analyze proficiency samples for all the parameters certified by the state of Arizona for drinking water and waste water. If you are not receiving all the required sample vials or if you are receiving additional sample vials that are not being tested, please contact your laboratory licensing consultant. We will make the necessary adjustment.

15. This is an update regarding the status of field methanolic preservation of soils for volatile organic analysis: The Arizona Environmental Laboratory Advisory Committee (ELAC) has approved the draft method generated by the ELAC technical subcommittee and at present is being reviewed by Arizona Department of Environmental Quality (ADEQ) staff. Following the approval by ADEQ, the field methanolic preservation will be mandated for all volatile organic analyses in soil. After the approved method has been issued by ADEQ, the Arizona Department of Health Services will offer training in the field preservation technique.
16. We thank the readers for responding to Item #7, Update #12, regarding the test methods. We will forward the information to ADEQ.

17. AWWA teleconference on "Source Water Protection: An Ounce of Prevention" is being held on August 3, 1995 at Phoenix Fire Training Academy, 2430 South 22nd Avenue, Phoenix, Arizona. Call (602) 241-1770 for more information.
18. If you have any questions regarding the Updates, please call Prabha Acharya, program manager, Technical Resources and Training, at the above numbers.

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